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This report describes and summarizes the activities during the second year of the program named above. The activities described are part of a continuing 5-year program.

The purpose of the program is to establish a quality assurance data base for ambient air monitoring in specified geographical areas around present and proposed energy development projects, and to provide technical assistance to enable existing monitoring networks to achieve a high level of data quality. An initial on-site review of 18 laboratories and associated field sites was completed during the first year. Additional laboratories and field sites were evaluated during the second year. Regularly scheduled laboratory performance surveys are being carried out for the analysis of sulfate, nitrate, SO_2, NO_2, and CO and for weight measurements and high volume flow rate. Approximately 10% of the analysis performed by a specified laboratory for metals collected in high volume filters are being repeated in the Rockwell laboratory. Quarterly field audits are being conducted at specified monitoring sites. Technical assistance has been provided to participating monitoring groups, as requested by the Project Officer.

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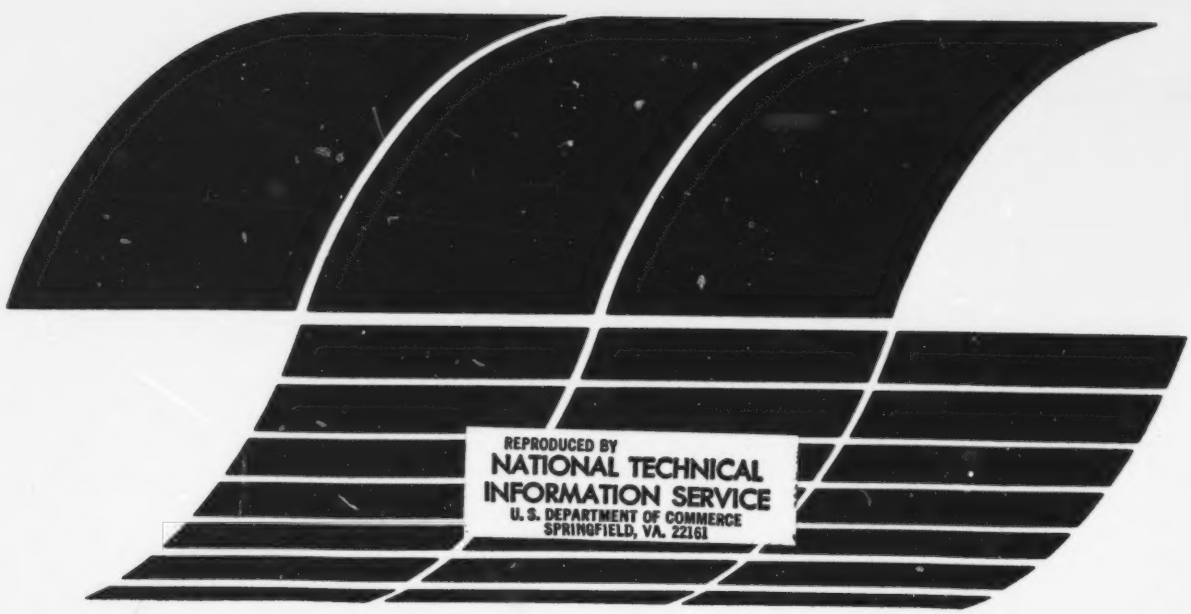

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QUALITY ASSURANCE IN SUPPORT OF ENERGY RELATED MONITORING ACTIVITIES

Annual Report No. 2

by

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DISCLAIMER

This report has been reviewed by the Environment Monitoring and Support Laboratory, U.S. Environmental Protection Agency, and approved for publication. Mention of trade names or commercial products does not constitute endorsement or recommendation for use.

ABSTRACT

This report describes and summarizes the activities and achievements of Rockwell International's Environmental Monitoring & Services Center (EMSC) during the second year of the program entitled, Quality Assurance in Support of Energy Related Monitoring Activities. The activities cover generally the period from September 1977 to September 1978.

The purpose of the program is to establish a quality assurance data base for ambient air monitoring in specified geographical areas around present and proposed energy development projects, and to provide technical assistance to enable existing monitoring networks to achieve a high level of data quality. A goal of the program is to enable the government to utilize and compare air monitoring data from diverse sources for future study and planning purposes by providing information concerning data quality from the individual monitoring networks.

A major change in the program occurred in September 1978, when the number of participants was greatly expanded to include laboratories and sites located in the Ohio and Tennessee Valleys, and also in several eastern states in Region III. An initial Quality System Audit of the new participants has been started, and detailed evaluation reports containing list of recommendations for eliminating observed deficiencies will be submitted to EPA.

Regularly scheduled Quality Control Reference Sample Audits are being continued for the analysis of sulfate, nitrate, SO_2, NO_2, and CO, and for weight measurement and high volume flow rate. In these surveys reference samples or devices are submitted to participating laboratories and their results are

compared with those obtained by Rockwell. Performance results for the first two years of the program have remained essentially constant for sulfate, nitrate, SO_2, and CO. The results for CO are noteworthy for their consistently high quality. Results for NO_2 have shown significant improvement, and in the last three surveys in 1978 the level of agreement between the laboratories and Rockwell has been generally very high. Improvements in performance have also been observed in the weighing and high volume surveys.

An interlaboratory comparison study involving Rockwell and another laboratory to determine the causes for the large differences in the analysis of metals in TSP samples which had been observed at the beginning of the program was completed in 1978. The differences were determined to be caused primarily by differences in extraction procedures. The laboratory has adopted the same procedure used by Rockwell, and subsequent analyses have shown major improvement in the level of agreement obtained in the analysis of copper, iron and manganese.

Quarterly Calibration System Audits are being continued at specified monitoring sites. Known concentrations of pollutants are delivered to each continuous analyzer, and the observed response is compared with that predicted by the agency's calibration. Procedures have been developed to assure reliable performance by the audit devices and standards. Additional checks on the equipment and procedures are made by means of quarterly audits performed in the EPA/EMSL laboratory at Research Triangle Park, North Carolina.

Approximately 150 Calibration System Audits have been completed thus far, and audit reports have been submitted to EPA. Evaluation procedures for reporting on individual and collective performance have undergone several changes during the past year. A major achievement of the program has been the remarkable

improvement in the calibration accuracy observed for several pollutants, especially NO, NO_x, and NO_2. Some problems with regard to the calibration of ozone analyzers are still evident.

Technical assistance has been provided to participating agencies, as requested by the Project Officer. Much of the assistance is given informally during site visits. Assistance was also provided concerning specific laboratory and calibration problems during special visits by Rockwell personnel to the participating agencies. A brief description of these and other types of technical assistance is given in the report.

This report was submitted in fulfillment of Contract No. 68-02-2412 by Rockwell International Environmental Monitoring & Services Center under the sponsorship of the U.S. Environmental Protection Agency. Work under this contract is scheduled to continue in the west until July 13, 1981, and in the east until September 13, 1983.

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SECTION 1

INTRODUCTION

This report describes and summarizes the activities and achievements of Rockwell International Environmental Monitoring & Services Center during the second year of the Contract No. 68-02-2412, entitled Quality Assurance in Support of Energy Related Monitoring Activities. The effective date of the contract was July 13, 1976, and the program is scheduled to be completed on July 13, 1981.

On March 24, 1977, the contract was enlarged to include additional Quality System Audits and Calibration System Audits of sites located in Colorado, Utah, and Montana. On August 9, 1977, the contract was further expanded to include Quality System Audits of seven water quality field stations of the U.S. Geological Survey in the states of Arizona, Colorado, Montana, New Mexico, North Dakota, Utah, and Wyoming.

A major modification to the contract was put into effect September 13, 1978, when the program was expanded to include laboratories and sites located in the Ohio and Tennessee Valleys, and also in several eastern states in Region III. At the time of the modification, the work for Region VIII, which was initiated on March 24, 1977, as described above, was extended for an additional 12 months.

The activities described in this report cover generally the period from September 1977 to September 1978. For convenience, we refer to the Western Q.A. Program and the Eastern Q.A. Program to describe the work according to the geographical location of the participating groups. The Eastern Q.A.

Program includes groups in the Ohio and Tennessee Valleys, and in the Region III states. In the period covered by this report, the overwhelming majority of the work was performed with groups comprising the original Western Q.A. Program.

The purpose of the Eastern and Western Quality Assurance Programs is to develop and implement a quality assurance program for use by networks monitoring air quality around present and proposed energy development projects. A goal of the program is to enable the government to utilize and compare air monitoring data from diverse sources for future study and planning purposes by providing information concerning data quality from the monitoring networks. A secondary goal is to provide technical assistance to the participating groups to help them achieve a high level of performance. In the west the energy projects are located in the states of Montana, South Dakota, North Dakota, Wyoming, Utah, Colorado, Arizona, Nevada, New Mexico, and California. In the east the energy projects are located in the states of Kentucky, West Virginia, Illinois, Ohio, Pennsylvania, and Indiana. Additional groups, which are not necessarily involved in energy development projects, are located in Delaware, Maryland, Virginia, and the District of Columbia, and are also associated with the Eastern Q.A. Program.

This Annual Report is written as a general overview of performance and progress thus far. In order to keep this report within manageable size, the reader will oftentimes be directed to specific reports, submitted to EPA, should greater detail be required.

The Eastern and Western Quality Assurance Programs are divided into four task areas involving:

1. Quality System Audits - an initial on-site review of laboratories and field sites;

2. Quality Control Reference Sample Audits - a sample submissions audit program to the laboratories;

3. Calibration System Audits - on-site field calibration audits; and

4. Technical assistance to the laboratories, as required.

All the work carried out by Rockwell in connection with these tasks is summarized in the following sections. Section 2 describes the Quality System Audits, Section 3 discusses the results of the Quality Control Reference Sample Audits and an interlaboratory comparison study; Section 4 summarizes Calibration System Audit results; Section 5 describes technical assistance given to the various agencies; and Section 6 outlines the plans for next year.

SECTION 2

QUALITY SYSTEM AUDITS

All Quality System Audits in the Western Q.A. Program were completed during the first year of the contract and are described in the first annual report. Table 1 lists the groups scheduled to be evaluated for the Eastern Q.A. Program. The list includes all the groups who have agreed to participate as of December 1978. Additional groups have been contacted by EPA and they will be added to the list if the necessary approvals can be secured.

Preliminary meetings with some of the eastern participants were held in Alexandria, Philadelphia, and Chicago in June and in August 1978. The Project Officer, the Program Manager, and the EPA Q.A. Coordinators for Regions III and V were in attendance at these meetings, which were held for the purpose of informing the participants about the goals, organization, mechanics, and schedules of the Eastern Q.A. Program.

As with the Western Q.A. agencies, all the groups in Table 1 will be visited, and the laboratories and field sites will be evaluated by Rockwell personnel. The same questionnaires used previously in the west were mailed to the eastern groups prior to the Rockwell visit in order to expedite the evaluation interview. The first four groups in Table 1 were visited by the Program Manager in August 1978. The rest of the groups will be visited at a later time.

A detailed report covering the Quality System Audits will be made for each group, and each group will be given the opportunity to comment upon the preliminary draft of the report. When appropriate, these comments will be

TABLE 1. LIST OF AIR MONITORING GROUPS PARTICIPATING IN EASTERN QA PROGRAM

Group	Headquarters	Air Monitoring Site Locations to be Audited
District of Columbia	Washington, DC	Washington, DC *
State of Virginia	Richmond, VA	Richmond*, Norfolk*, Hampton*
Fairfax County	Fairfax, VA	McLean*
City of Alexandria	Alexandria, VA	Alexandria*
State of Maryland	Baltimore, MD	Baltimore*, Washington* DC (Maryland suburbs)*
County of Baltimore	Baltimore, MD	Baltimore*
City of Baltimore	Baltimore, MD	Baltimore*
State of Delaware	Wilmington, DE	Wilmington*
State of Pennsylvania	Harrisburg, PA	Various locations in state
City of Philadelphia	Philadelphia, PA	Philadelphia
Allegheny County	Pittsburgh, PA	Pittsburgh
State of West Virginia	Charleston, WV	Charleston, Wheeling
Ohio Edison	Akron, OH	Southern Ohio
Pennsylvania Electric	Johnstown, PA	Indiana, PA
State of Indiana	Indianapolis, IN	Southern Indiana
City of Cincinnati	Cincinnati, OH	Cincinnati
Regional Air Pollution Control Agency	Dayton, OH	Dayton

* These sites are not energy related but were added on to the program for convenience.

noted and used in preparing the final report, copies of which will be sent to the EPA.

SECTION 3

QUALITY CONTROL REFERENCE SAMPLE AUDITS

Regularly scheduled Quality Control Reference Sample Audits have been carried out for the following analyses or measurements;

1. Sulfate/Nitrate Analysis
2. SO_2 Analysis
3. NO_2 Analysis
4. CO Analysis
5. Weighing Performance
6. High Volume Flow Rate Measurement.

In addition to the laboratory surveys, an interlaboratory comparison study has been carried out with one laboratory in which approximately 75 filters were analyzed by the laboratory and Rockwell for trace metals and the results compared.

Table 2 lists all the surveys performed in the first two years of the program by date. Participating laboratories in each survey are indicated by a check (✓) mark.

For the chemical analysis survey (1-4 in the above list), Rockwell obtains from commercial vendors, either directly or through EPA, multiple sets of the appropriate samples which are then submitted for analysis to participating laboratories. Ten replicate samples are first analyzed at Rockwell, and the mean value of the analyses is by definition the "true" value with which the results of all the participants are compared.

To assure the correctness of the Rockwell "true" value, several internal

TABLE 2. LIST OF INTERLABORATORY PERFORMANCE SURVEYS AND PARTICIPANTS(*)

Agency	Survey and Date by Quarter													
	$SO_4^=/NO_3^-$							SO_2						
	4/76 #	1/77 #	2/77 #	3/77 #	4/77	1/78	2/78	4/76 #	2/77 #	3/77 #	4/77	1/78	2/78	3/78
Arizona State	✓†	✓†	✓†	✓†	✓†	✓	✓	✓		✓	✓	✓	✓	✓
Colorado State	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Energy and Environmental Resource Consultants							✓						✓	✓
Lockheed									✓					
Montana State	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
New Mexico State	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
North Dakota State	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓
Northern Testing Lab	✓	✓	✓	✓	✓	✓		✓	✓	✓	✓	✓		
Northrop Services Inc.											✓		✓	✓
South Dakota State						✓	✓	✓	✓	✓	✓	✓	✓	✓
Ute Research			✓	✓	✓	✓	✓							
Wyoming State								✓	✓	✓	✓	✓	✓	✓
Yellowstone County								✓	✓	✓	✓	✓	✓	✓

(*) A check (✓) means that the agency participated in the survey.
(†) $SO_4^=$ only
(#) Summary data for these surveys in first annual report.

TABLE 2 (Continued)

Agency	Survey and Date by Quarter																	
	NO_2							CO							Weighing		Hi-Vol	
	1/77 #	2/77 #	3/77 #	4/77	1/78	2/78	3/78	4/76 #	1/77 #	3/77 #	4/77	1/78	2/78		1/77 #	1/78	1/77 #	1/78
Albuquerque								✓	✓	✓	✓	✓	✓		✓	✓	✓	✓
Arizona State	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓		✓	✓	✓	✓
C-a															✓	✓	✓	✓
C-b															✓	✓	✓	✓
Colorado State	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓		✓	✓	✓	✓
Energy and Environmental Research consultants						✓	✓											✓
Lawrence Radiation Labs																	✓	
Montana State	✓	✓	✓	✓	✓	✓	✓		✓	✓	✓	✓	✓		✓	✓	✓	✓
Montana Power Co.																	✓	✓
New Mexico State	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓		✓	✓	✓	✓
No. Dakota State	✓	✓	✓	✓	✓	✓	✓								✓	✓	✓	✓
Northern Testing Lab	✓	✓	✓	✓	✓										✓	✓		
So. Dakota State	✓	✓	✓	✓	✓	✓	✓								✓	✓	✓	✓
Ua/Ub															✓	✓	✓	✓
Ute Research															✓	✓	✓	✓
Utah State								✓	✓	✓	✓	✓	✓		✓	✓	✓	✓
Wyoming State	✓	✓	✓	✓	✓	✓	✓								✓	✓	✓	✓
Yellowstone County								✓							✓	✓	✓	✓

(*) A check (✓) means that the agency participated in the survey.
(#) Summary data for these surveys in first annual report.

and external quality control procedures are being used. Internal quality control procedures in the Rockwell laboratory include comparison of analyses of replicate samples, comparison of every new stock standard solution with the old one, duplicate calibration curves before and after analysis, analysis of quality control standards every 10 samples, and maintenance of routine quality control charts on calibration parameters to establish laboratory control limits. External quality control is provided by comparing the Rockwell "true" value with the mean value obtained through replicate sample analysis either by the QAB EMSL-RTP laboratory or by the vendor. This comparison is included in each report.

The evaluation of laboratory performance is done in several ways. The most direct way is to compare the concentration determined by each laboratory with the "true" value as determined by Rockwell. Linear graphs are prepared in which the laboratory values are plotted against the "true" values. The slope, intercept, and correlation coefficient for each laboratory line are then tabulated as indices of performance. Perfect agreement between a laboratory and Rockwell for all analyses results in a straight line with unit slope, zero intercept, and a correlation coefficient equal to 1.0000.

The slope of the line is a good measure of overall analytical accuracy, provided that the intercept is small compared to the concentrations being analyzed. For example, if the intercept is small, a slope of 1.05 implies a level of agreement with Rockwell of 5%.

If the slope is near 1.0, a large intercept indicates a bias in the analysis which might be caused by such errors as incorrect blank corrections or contaminations in the water supply. The magnitude of the intercept must

be evaluated by comparison with the sample size. For example, if the sample size is 10 $\mu g/m^3$, an intercept of 1 $\mu g/m^3$ represents a 10% bias.

A second method used for evaluating laboratory performance is to calculate average percent differences between the laboratory analysis values and the "true" values. The average percent differences as well as the standard deviation of the individual percent differences are tabulated by laboratory for each quarterly audit as measures of analytical error. The calculations are explained in detail in the proposed EPA regulations which appeared in the Federal Register in August 1978 as EPA 40 CFR Part 58, "Air Quality Surveillance And Data Reporting;" Appendix A - Quality Assurance Requirements for State and Local Air Monitoring Stations (SLAMS) (1). The major difference between our procedure and that described in the Federal Register is that the latter specifies 0.20 and 0.80 µg/ml for the concentration of the audit samples for both SO_2 and NO_2, while in the current program the concentration of the audit samples cover a much wider range. However, we understand that Appendix A is being changed and the final regulations will be promulgated sometime in 1979. Another difference from the EPA regulations is that our program includes audits of $SO_4^=$ and NO_3^- analysis, neither of which is required by the proposed regulations.

A third method used to evaluate laboratory performance is to determine whether each laboratory analysis results falls within arbitrarily defined concentration ranges denoted as "sample range" and "target range". The "sample range" is intended to describe the variability in the analysis of presumably identical samples within one laboratory but at various times and under various conditions, i.e., "normal laboratory operation". The "target range" is intended to bracket an acceptable range of variability among different laboratories and is larger than the "sample range".

Definitions of "sample range" and "target range" have not remained the same throughout the program. For most of the surveys the "sample range" and the "target range" are defined as $\bar{R} \pm 3\sigma$ and $\bar{R} \pm 5\sigma$, respectively, where $\bar{R}$ is the "true" value (i.e., the mean value of Rockwell's analyses) and σ is the standard deviation of Rockwell's replicate analyses. For CO the standard deviation σ is extremely small so that a more practical definition is required. For CO the "sample range" and "target range" were defined at various times as $\bar{R} \pm r$ where r was either a constant ppm value (e.g., $r = \pm 0.5$ ppm), or a constant percent of $\bar{R}$ (e.g., $r = \pm 4\%$). Of course, the value of r was not the same for the sample range and for the target range.

Because the definitions of range depend on the performance of the Rockwell laboratory through the experimentally determined value of σ, great care should be exercised in evaluating performance by means of the range criteria, particularly when comparing results from different surveys. The "sample range" and "target range" criteria are most useful for comparing the performance of different laboratories within the same survey. Because of this limitation, no discussion will be given inthis report in terms of the range criteria. The interested reader should consult the individual Quality Control Reference Sample Audit (Interlaboratory Performance Survey) reports submitted to EPA.

Each type of survey will be discussed eaparately below.

Sulfate/Nitrate Analysis

The sulfate/nitrate performance survey requires participating laboratories to analyze a set of four to six filter strips spiked with varying

amounts of sulfate and nitrate ions. At least 10 sets of filters presumed to be identical to those analyzed by the laboratories are analyzed by the Rockwell Chemistry Laboratory using the methylthymol blue procedure for $SO_4^=$ and the copperized cadmium reduction method for NO_3^-. In the interpretation of results no bias or adjustment is made for the fact that laboratories included in the surveys use methods different from Rockwell. All laboratories use EPA accepted methods which give presumably equivalent results, making such adjustments unnecessary.

Tables 3 and 4, taken from the second quarter 1978 report, list average percent difference $\bar{d}_j$, standard deviation S_j, and the linear parameters associated with each laboratory for all surveys to date. The average percent difference $\bar{d}_j$ is obtained by first calculating the percent difference for each individual sample using the equation

$$d_i = \left(\frac{Y_i - X_i}{X_i} \right) 100 \qquad (1)$$

where Y_i and X_i are the concentrations determined by a laboratory and by Rockwell, respectively. The value of $\bar{d}_j$ is simply the average of all the d_i's for each laboratory and S_j is the standard deviation of the d_i's. To preserve anonymity, the identities of the laboratories are not shown. (There is no correlation between the identity codes in the tables of this report. Thus Agency A in one table is not necessarily the same as Agency A in another table.)

The composite data in Tables 3 and 4 were obtained by combining results of all samples and all laboratories and are used to evaluate overall performance. Figure 1 shows graphs of $\bar{d}_j$ and $\bar{d}_j \pm S_j$ for the composite data as a

TABLE 3. SUMMARY OF AGENCY PERFORMANCE FOR $SO_4^=$ SURVEYS

Agency Code	Quarter/ Year	Avg % Diff. $\bar{d}_j$	Std. Dev. S_i	Slope	Intercept $\mu g/m^3$
A	2/77	7.3	4.5	1.005	0.333
	3/77	6.8	3.5	1.042	0.194
	4/77	5.4	5.8	1.009	0.843
	1/78	4.9	15.5	1.175	-0.383
	2/78	12.5	7.2	1.036	0.287
B	1/77	2.5	7.9	1.030	0.075
	2/77	10.7	9.7	1.046	0.433
	3/77	3.2	3.2	1.011	0.149
	4/77	-3.0	4.7	0.950	0.690
	1/78	6.3	7.8	1.010	0.524
C	2/78	29.6	74.2	0.801	1.537
D	4/76	-16.2	18.0	0.926	0.075
	1/77	5.9	3.7	1.031	0.459
	2/77	-13.3	9.5	0.755	0.982
	3/77	-18.7	36.5	1.093	-0.758
	4/77	-6.4	10.1	0.838	1.355
	1/78	-23.6	59.1	1.326	-1.797
	2/78	6.1	4.2	0.962	1.209
E	4/76	-5.6	21.4	1.054	-0.012
	1/77	9.0	5.0	1.087	0.147
	2/77	-0.5	12.6	0.869	0.556
	3/77	-20.8	31.1	0.543	0.735
	4/77	-2.9	15.9	0.960	-0.225
	1/78	-8.8	22.3	1.097	-0.529
	2/78	-3.4	9.9	1.022	-0.041

(continued)

TABLE 3 (Continued)

Agency Code	Quarter/ Year	Avg % Diff. $\bar{d}_j$	Std. Dev. S_j	Slope	Intercept $\mu g/m^3$
F	4/76	34.7	54.1	0.810	1.066
	1/77	12.6	14.3	1.351	-2.973
	2/77	-14.6	19.2	1.102	-1.286
	3/77	-19.0	33.7	0.848	-0.301
	4/77	-1.5	26.5	1.223	-3.126
	1/78	-6.5	12.4	1.039	-0.340
	2/78	38.2	51.7	0.972	1.393
G	4/76	** 119.2	123.3	0.643	4.191
	1/77	12.3	8.5	1.075	0.875
	2/77	** 131.6	31.3	1.860	2.272
	3/77	3.9	13.8	0.948	0.886
	4/77	0.6	7.2	1.039	-0.392
	1/78	-1.1	13.1	1.061	-0.055
	2/78	0.3	5.9	1.058	-0.144
H	4/76	-9.3	12.4	1.036	-0.194
	1/77	-4.9	4.0	0.895	0.718
	2/77	-25.9	18.5	0.913	-0.565
	3/77	9.9	4.6	1.045	0.398
	4/77	3.8	4.7	1.049	-0.205
	1/78	-13.4	13.1	0.977	-0.353
	2/78	6.6	2.8	1.027	0.148
I	1/78	-1.9	17.0	1.073	-0.133
	2/78	-0.4	13.8	1.005	0.122
EPA/QAB	4/76	-0.1	1.9	1.032	-0.114
	1/77	3.3	2.3	1.053	-0.190
	2/77	-1.2	8.3	0.946	0.290

** Laboratory data not used for computing composite results.

(continued)

TABLE 3 (Continued)

Agency Code	Quarter/ Year	Avg % Diff. $\bar{d}_j$	Std. Dev. S_i	Slope	Intercept $\mu g/m^3$
EPA/QAB (cont'd)	3/77	2.8	3.7	1.039	0.001
	1/78	-2.9	15.1	1.093	-0.402
	2/78	6.1	3.9	1.055	0.039
Composite	4/76	0.7	32.6	0.971	0.164
	1/77	5.9	9.3	1.075	-0.127
	2/77	-5.3	16.9	0.948	0.106
	3/77	-4.0	23.0	0.946	0.163
	4/77	-0.6	12.1	1.010	-0.151
	1/78	-5.5	23.9	1.095	-0.385
	2/78	10.8	31.7	0.993	0.505

TABLE 4. SUMMARY OF AGENCY PERFORMANCE FOR NO_3^- SURVEYS

Agency Code	Quarter/ Year	Avg % Diff. $\bar{d}_j$	Std. Dev. S_i	Slope	Intercept $\mu g/m^3$
A	2/77	29.4	6.3	1.344	-0.060
	3/77	0.1	8.6	0.902	0.191
	4/77	5.9	8.4	0.923	0.244
	1/78	7.3	1.3	1.075	-0.014
	2/78	-2.1	10.7	1.091	-0.260
B	4/76	63.1	88.8	0.941	0.579
	1/77	-11.3	4.5	0.845	0.203
	2/77	-18.6	9.0	0.878	-0.070
	3/77	6.7	11.0	0.997	0.098
	4/77	-0.5	17.5	1.011	0.084
	1/78	15.5	38.2	0.818	0.473
C	2/78	4.7	84.4	0.963	0.007
D	1/78	2.7	18.1	1.159	-0.148
	2/78	2.7	6.1	1.082	-0.191
E	4/76	-0.9	5.9	1.055	-0.084
	1/77	-0.1	22.2	1.025	-0.348
	2/77	** 157.3	350.2	0.505	2.333
	3/77	4.4	12.3	0.958	0.143
	4/77	-41.8	6.2	0.507	0.092
	1/78	-2.5	17.6	1.074	-0.135
	2/78	-6.4	20.5	1.126	-0.394
F	4/76	35.7	42.0	1.027	0.310
	1/77	20.8	13.1	1.012	0.956
	2/77	5.7	17.0	0.883	0.349

** Laboratory data not used for computing composite results.

(Continued)

TABLE 4 (Continued)

Agency Code	Quarter/ Year	Avg % Diff. $\bar{d}_j$	Std. Dev. S_i	Slope	Intercept $\mu g/m^3$
F (cont'd)	3/77	-16.5	10.2	0.910	-0.037
	4/77	9.5	6.0	1.016	0.111
	1/78	6.1	5.9	1.049	-0.001
	2/78	9.6	2.4	1.098	-0.038
G	4/76	** 5885.2	7835.4	-2.180	44.680
	1/77	27.3	21.3	1.050	1.034
	2/77	** 105.1	6.4	2.085	0.013
	3/77	22.2	7.4	1.184	0.078
	4/77	12.8	16.2	1.000	0.181
	1/78	2.3	3.2	1.035	-0.015
	2/78	-3.6	12.5	1.079	-0.244
H	4/76	14.5	11.6	1.249	-0.203
	1/77	-11.2	4.3	0.878	0.018
	2/77	-8.6	10.8	0.914	-0.005
	3/77	15.7	4.6	1.116	0.081
	4/77	21.9	12.9	1.051	0.211
	1/78	14.3	4.8	1.108	0.035
	2/78	4.2	8.6	1.137	-0.250
I	1/78	0.6	10.7	1.061	-0.069
	2/78	-10.1	14.0	1.043	-0.343
EPA/QAB	4/76	1.4	0.7	1.018	-0.002
	1/77	9.3	4.2	1.050	0.167
	2/77	-8.0	2.8	0.948	-0.040
	3/77	1.3	2.3	1.036	-0.031

** Laboratory data not used for computing composite results.

(continued)

TABLE 4 (Continued)

Agency Code	Quarter/ Year	Avg % Diff. $\bar{d}_j$	Std. Dev. S_i	Slope	Intercept $\mu g/m^3$
EPA/QAB	1/78	6.4	2.6	1.058	-0.014
(cont'd)	2/78	-9.4	22.9	1.103	-0.391
Composite	4/76	24.6	48.9	1.058	0.120
	1/77	4.3	19.5	0.977	0.338
	2/77	0.0	19.4	0.993	0.035
	3/77	4.8	14.0	1.015	0.075
	4/77	1.3	23.5	0.918	0.154
	1/78	5.6	14.5	1.048	0.012
	2/78	-1.2	28.8	1.080	-0.234

function of time. The curves for $\bar{d}_j \pm S_j$ describe a band of one standard deviation above and below $\bar{d}_j$. The band is very roughly ± 20% for both sulfate and nitrate. The significance of the band is that approximately 2/3 of all the laboratory analyses agree with the "true" values as determined by Rockwell to within ± 20%.

Ideally, the composite values of $\bar{d}_j$ should oscillate around zero, and the differences from zero are probably related to the population size and to the accuracy and precision of both Rockwell and the participating laboratories. Exactly what this relationship is and how much variation around zero might be expected are not clear at this time.

Although the above statistical treatment is not mathematically rigorous, it does demonstrate that laboratory performance has remained essentially constant over the lifetime of the program. The lack of dramatic improvement is not completely unexpected since most of the laboratories have participated in similar EPA surveys prior to the beginning of the Western Q.A. Program.

SO_2 Analysis

SO_2 samples consist of standard ampules containing freeze-dried solutions of sodium sulfite and tetrachloromercurate. The "true" value for each sample is obtained by the Rockwell laboratory by analysis of at least 10 replicate samples. The usual quality control procedures were used for each analysis.

Table 5, taken from the third quarter 1978 report, summarizes the SO_2 results since the beginning of the program. Composite results are shown graphically in Figure 1. Approximately 2/3 of all the analyses agree with

TABLE 5. SUMMARY OF AGENCY PERFORMANCE FOR SO_2 ANALYSIS

Agency Code	Quarter/ Year	Avg % Diff. $\bar{d}_j$	Std. Dev. S_j	Slope	Intercept $\mu g/m^3$
A	4/76	-24.4	19.3	0.949	-8.972
	1/77	-28.3	35.9	1.013	-14.095
	3/77	-18.7	8.4	0.910	-5.987
	4/77	-14.2	50.2	1.292	-18.727
	1/78	6.1	3.7	1.000	3.730
	2/78	8.6	4.6	1.030	3.216
	3/78	3.2	3.3	1.066	-2.673
B	4/76	-25.3	38.5	1.100	-15.615
	3/77	1.5	10.9	0.862	10.998
	4/77	-33.7	46.9	0.391	8.718
	1/78	9.8	18.5	0.974	4.883
	2/78	-4.1	20.1	1.142	-8.911
	3/78	-18.7	31.7	1.099	-13.526
C	4/76	9.5	3.9	1.119	-0.733
	1/77	-2.6	3.7	1.005	-1.408
	3/77	2.4	7.0	1.032	0.728
	4/77	10.2	9.3	1.157	-1.859
	1/78	1.6	7.1	0.969	1.710
	2/78	21.0	2.3	1.204	0.725
	3/78	0.6	8.7	1.139	-8.132
D	4/76	1.7	9.8	0.963	1.689
	1/77	-2.5	2.9	0.977	0.287
	3/77	-10.5	6.3	0.923	-0.643
	4/77	-58.9	26.9	0.776	-19.789
	1/78	2.1	3.7	0.985	1.765

(continued)

TABLE 5 (Continued)

Agency Code	Quarter/ Year	Avg % Diff. $\bar{d}_j$	Std. Dev. S_j	Slope	Intercept $\mu g/m^3$
D (cont'd)	2/78	3.8	2.2	1.041	0.135
	3/78	5.8	7.2	1.074	-2.422
E	4/76	4.7	10.2	0.980	2.488
	1/77	-7.6	5.5	1.023	-8.276
	3/77	-5.6	3.3	0.937	1.208
	4/77	-5.3	8.3	0.993	-1.405
	1/78	-2.8	11.4	0.977	1.781
	2/78	-2.2	3.2	0.999	-0.918
	3/78	4.9	5.5	1.012	1.439
F	4/76	-11.4	6.2	0.876	-0.117
	1/77	-23.3	12.2	0.668	7.490
	3/77	-4.5	4.6	0.888	4.731
	4/77	0.2	13.6	0.905	4.054
	1/78	1.1	7.1	0.950	3.795
	2/78	-20.0	25.2	0.286	34.284
	3/78	9.5	10.8	0.937	9.104
G	4/76	23.9	29.6	1.348	-10.974
	1/77	-7.7	18.5	1.097	-8.223
	3/77	-12.7	11.3	1.018	-10.473
	4/77	-5.2	9.4	0.764	12.145
	1/78	-0.9	4.5	0.983	1.471
	2/78	-19.6	18.6	0.533	25.314
	3/78	7.6	4.2	1.053	0.645
H	4/76	-2.7	7.0	1.062	-5.954
	1/77	0.4	11.0	0.931	3.010
	3/77	-24.7	25.6	0.995	-12.075

(continued)

TABLE 5 (Continued)

Agency Code	Quarter/ Year	Avg % Diff. $\bar{a}_j$	Std. Dev. S_j	Slope	Intercept $\mu g/m^3$
H (cont'd)	4/77	2.8	10.1	0.992	0.492
	1/78	-2.4	30.7	1.058	-8.848
	2/78	-33.3	27.1	1.019	-20.119
	3/78	8.1	0.0	***	***
I	2/78	-4.8	15.5	1.129	-9.250
	3/78	-5.7	5.7	0.936	-0.524
J	4/77	-47.7	33.7	0.407	15.000
	2/78	1.9	3.9	1.043	-1.910
	3/78	7.8	5.4	1.074	-0.620
K	4/76	1.6	3.1	1.011	0.597
	1/77	-4.0	3.1	1.000	-2.484
	3/77	-11.9	10.2	0.978	-4.838
	4/77	-16.4	16.5	1.033	-10.078
	1/78	-5.5	12.8	1.003	-1.509
EPA/QAB	4/76	4.6	10.3	0.974	2.855
	1/77	1.1	6.9	0.945	3.320
	3/77	-9.6	9.6	0.980	-3.378
	4/77	-0.6	4.1	0.999	0.146
	3/78	11.5	4.1	1.092	0.724
Composite	4/76	-2.0	21.8	1.038	-3.474
	1/77	-7.8	16.0	0.962	-2.264
	3/77	-9.4	13.0	0.952	-1.973
	4/77	-15.4	31.7	0.883	-1.028
	1/78	1.0	12.9	0.989	0.975
	2/78	-4.6	20.5	0.943	2.256
	3/78	2.6	13.8	1.048	-1.598

*** Data based on a single sample analysis.

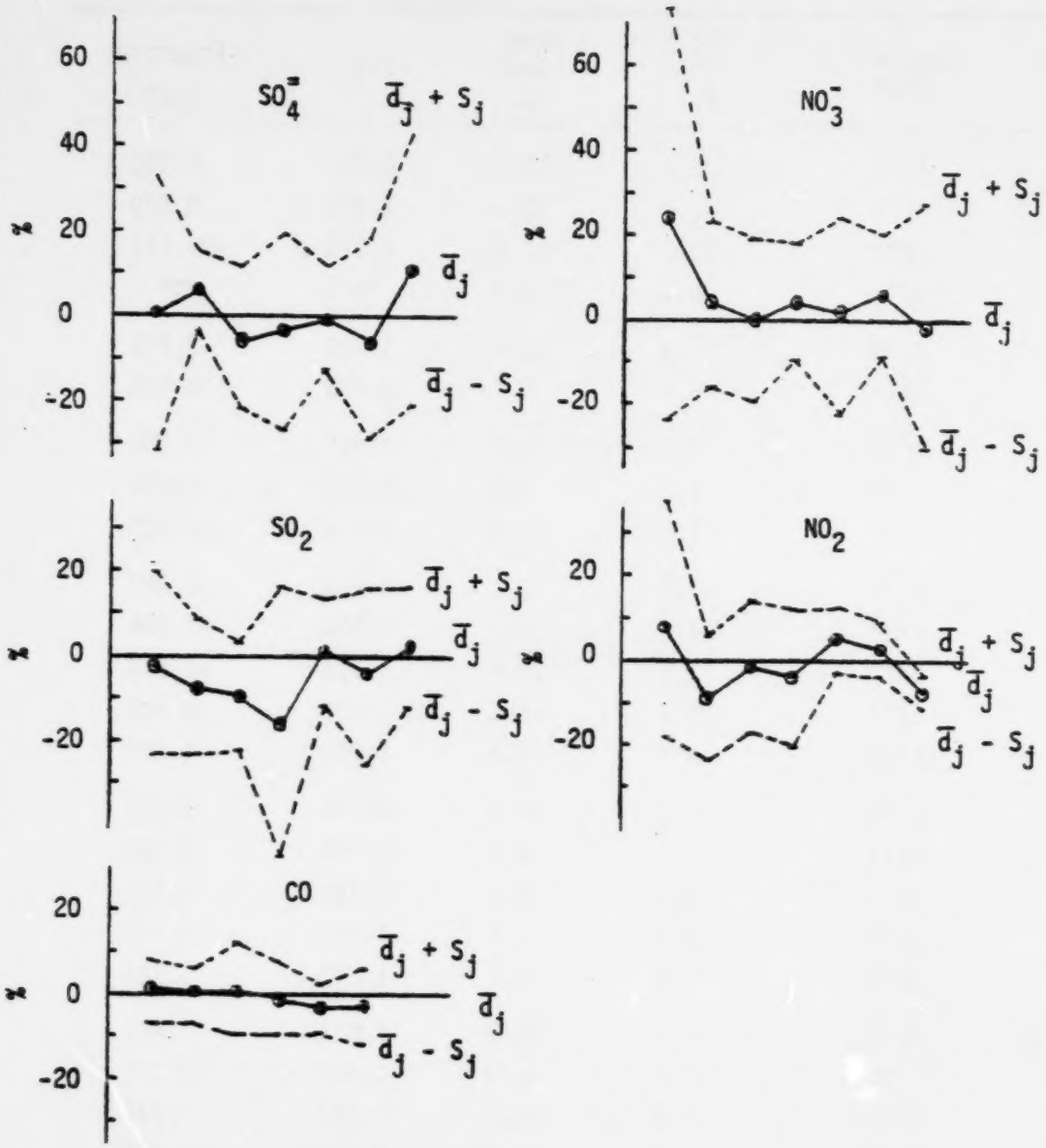


Figure 1. Composite quarterly performance evaluation for all surveys. Graphs show $\bar{d}_j$ and $\bar{d}_j \pm S_j$ in units of percent difference as a function of time since the beginning of the program. Each point is obtained by averaging all the analysis data for one given quarterly survey. The CO, SO_2, NO_3^- and $SO_4^=$ data commence in the fourth quarter of 1976. The NO_2 evaluation began in the first quarter of 1977.

the Rockwell analyses within the percentage limits defined by $\bar{d}_j \pm S_j$. For SO_2, the $\bar{d}_j \pm S_j$ band is approximately between + 16% and -23%. Thus the overall SO_2 performance is roughly comparable to that of $SO_4^=$ and NO_3^-. The composite $\bar{d}_j$ appears to show a small negative bias which might be explained by degradation of samples during the period between the analysis at Rockwell and the analysis at the other laboratories. In view of this and other uncertainties, this explanation is highly conjectural. A significant anomaly appears to exist in the fourth survey as indicated by the large values of $\bar{d}_j$ and S_j ($\bar{d}_j$ = -15.4%, S_j = 31.7%). For this particular survey, a sample homogeneity problem is suspected since at least three of the eleven laboratories reported very large negative differences. In addition, the first batch of analyses carried out at the Rockwell laboratory had to be discarded because of unusually high scatter, while a second batch appeared to give more reasonable results.

NO_2 Analysis

NO_2 samples consist of standard ampules containing solutions of sodium nitrite. The "true" value for each sample is obtained by the Rockwell laboratory by analysis of at least 10 replicate samples. The usual quality control procedures were used for each analysis.

Table 6, taken from the 3rd quarter 1978 report, summarizes the NO_2 results since the beginning of the program. Composite results are shown graphically in Figure 1. The results for NO_2 appear to be qualitatively different from those of $SO_4^=$, NO_3^-, and SO_2 in that the band defined by $\bar{d}_j \pm S_j$ is narrower, and there appears to be a genuine trend towards improved

TABLE 6. SUMMARY OF AGENCY PERFORMANCE FOR NO_2 ANALYSIS

Agency Code	Quarter/ Year	Avg % Diff. $\bar{d}_j$	Std. Dev. S_j	Slope	Intercept µg/ml
A	1/77	62.6	54.9	1.086	0.136
	2/77	-9.4	7.4	0.996	-0.025
	3/77	0.2	6.3	0.931	0.017
	4/77	-4.4	13.6	0.904	0.008
	1/78	7.0	9.7	0.992	0.022
	2/78	7.0	10.7	0.939	0.031
	3/78	-7.2	5.0	1.005	-0.033
B	1/77	-8.4	7.3	1.028	-0.033
	2/77	-10.2	8.9	1.000	-0.028
	3/77	-19.1	27.1	1.037	-0.052
	4/77	3.8	12.4	0.958	0.018
	1/78	6.3	6.4	1.004	0.018
	2/78	3.7	5.5	0.967	0.016
	3/78	-6.0	2.1	0.984	-0.019
C	1/77	-1.0	2.8	1.009	-0.004
	2/77	-6.1	4.4	0.987	-0.018
	3/77	0.1	9.5	0.820	0.048
	4/77	1.1	2.7	1.007	0.001
	1/78	8.1	4.6	1.042	0.012
	2/78	0.6	2.1	0.990	0.003
	3/78	-4.4	3.0	1.007	-0.021
D	2/78	0.6	3.5	0.970	0.009
	3/78	-6.0	1.9	0.915	0.010
E	1/77	7.4	6.9	0.961	0.031
	2/77	-13.5	11.9	0.976	-0.029
	3/77	4.0	6.1	0.964	0.019

(continued)

TABLE 6 (Continued)

Agency Code	Quarter/ Year	Avg % Diff. $\bar{d}_j$	Std. Dev. S_j	Slope	Intercept µg/ml
E (cont'd)	4/77	6.7	8.5	0.973	0.025
	1/78	8.8	5.6	1.020	0.022
F	1/77	19.8	18.4	1.261	-0.034
	2/77	-18.7	3.3	0.879	-0.020
	3/77	-12.4	7.4	0.941	-0.021
	4/77	-8.5	12.5	1.020	-0.025
	1/78	-7.2	2.3	0.926	-0.001
	2/78	10.4	7.5	0.996	0.028
	3/78	-7.0	7.3	0.969	-0.012
G	1/77	5.0	10.8	0.946	0.023
	2/77	-11.9	4.4	0.967	-0.029
	3/77	-0.5	6.8	0.933	0.014
	4/77	-2.7	2.5	1.003	-0.008
	1/78	10.3	7.5	1.018	0.028
	2/78	2.2	3.1	0.998	0.005
	3/78	-5.6	2.7	0.998	-0.022
H	1/77	-5.2	3.1	0.918	0.007
	2/77	-1.2	7.4	0.932	0.014
	3/77	5.6	21.4	0.841	0.050
	4/77	-26.1	37.3	1.077	-0.082
	1/78	2.3	5.3	0.987	0.010
	2/78	1.1	5.1	0.962	0.011
	3/78	-7.9	1.0	0.935	-0.006
I	1/77	5.8	8.6	0.981	0.018
	2/77	-0.2	40.5	0.763	0.051
	3/77	11.7	15.8	0.909	0.051
	4/77	-0.9	4.9	0.969	0.009

(continued)

TABLE 6 (Continued)

Agency Code	Quarter/ Year	Avg % Diff. $\bar{d}_j$	Std. Dev. S_j	Slope	Intercept µg/ml
I (cont'd)	1/78	9.0	7.1	1.017	0.023
	2/78	4.7	7.1	0.962	0.020
	3/78	-5.0	1.7	0.998	-0.034
EPA/QAB	1/77	-1.6	6.1	0.927	0.012
	2/77	-9.2	2.1	0.919	-0.003
	3/77	1.3	10.9	1.274	-0.074
	4/77	-2.5	3.6	0.924	0.015
	1/78	2.6	5.4	0.982	0.013
	2/78	-1.6	6.7	0.931	0.011
	3/78	-11.0	3.2	0.949	-0.024
Composite	1/77	9.4	27.6	1.013	0.017
	2/77	-8.9	14.7	0.935	-0.010
	3/77	-1.0	15.7	0.961	0.006
	4/77	-3.7	16.3	0.982	-0.004
	1/78	5.3	7.7	0.999	0.016
	2/78	3.2	6.7	0.968	0.015
	3/78	-6.8	3.8	0.973	-0.018

data quality. For the first four surveys the standard deviation S_j was 15% or greater, while for the last three surveys S_j decreased monotonically down to less than 4%. In the last three surveys in 1978, the level of agreement between the laboratories and Rockwell has been generally very high.

CO Analysis

CO samples are contained in 0.85 m^3 (30 ft^3) aluminum cylinders. Three different concentrations were submitted for analysis in each survey. Replicate analyses are performed in the Rockwell Quality Assurance Laboratory using a Bendix nondispersive infrared analyzer, and an NBS SRM cylinder containing 95.0 ppm CO in air is used as the standard. The "true" value is established by analysis of at least five cylinders of each concentration.

The results of all the CO surveys are for the most part very satisfactory. Table 7 shows summary data for all laboratories. Composite results are shown graphically in Figure 1. The standard deviation S_j has been generally less than 10% since the beginning of the program. Thus approximately 2/3 of the laboratories report CO concentration which agree with those determined by Rockwell to within 10% or better. As indicated in the first annual report, the good agreement between the various agencies and Rockwell is probably due to the fact that agencies use cylinders for calibration that contain CO at ambient levels and require no dilution. The CO concentrations supplied by vendors are apparently sufficiently reliable for this particular analysis.

TABLE 7. SUMMARY OF AGENCY PERFORMANCE FOR CO ANALYSIS

Agency Code	Quarter/ Year	Avg % Diff. $\bar{d}_j$	Std. Dev. S_j	Slope	Intercept ppm
A	4/76	5.1	5.7	1.073	-0.264
	1/77	5.4	2.7	1.052	-0.017
	2/77	4.0	4.2	1.030	0.042
	4/77	1.3	2.1	1.009	0.049
	1/78	-3.2	3.4	1.005	-0.602
	2/78	-0.6	2.6	1.003	-0.113
B	4/76	5.8	12.2	0.956	0.969
C	4/76	0.8	6.2	0.997	0.108
	1/77	-5.4	5.7	0.968	0.005
	2/77	-17.1	34.0	1.076	-2.213
	4/77	-0.7	10.9	0.989	0.026
	1/78	2.0	7.7	1.014	0.101
	2/78	-4.2	3.4	0.993	-0.454
D	4/76	-3.7	5.0	1.019	-0.621
	1/77	4.3	4.7	1.079	-0.493
	2/77	6.7	6.9	1.126	-0.584
	4/77	-1.4	1.8	0.985	0.016
	1/78	-5.0	3.0	0.973	-0.279
	2/78	-18.6	13.8	1.043	-3.532
E	4/76	-2.3	7.1	0.994	-0.138
	1/77	-1.0	4.7	0.979	0.146
	2/77	-1.1	10.2	1.022	-0.314
	4/77	-0.7	13.3	0.981	0.182
	1/78	-2.3	5.6	0.986	-0.081
	2/78	-0.2	8.5	0.999	0.016

(continued)

TABLE 7 (Continued)

Agency Code	Quarter/ Year	Avg % Diff. $\bar{d}_j$	Std. Dev. S_j	Slope	Intercept ppm
F	1/77	-2.9	5.8	0.939	0.319
	2/77	-0.4	3.8	0.985	0.086
	4/77	-3.6	4.4	0.995	-0.279
	1/78	-5.6	3.9	0.993	-0.529
	2/78	-0.5	2.8	0.972	0.324
G	4/76	8.2	7.2	1.033	0.431
	1/77	6.7	2.5	1.041	0.295
	2/77	6.5	8.1	1.032	0.348
	4/77	0.5	4.9	1.033	-0.197
	1/78	-7.7	3.8	0.957	-0.484
	2/78	10.7	7.8	1.005	1.510
EPA/QAB	4/76	0.5	1.7	1.002	-0.034
	1/77	-2.0	3.2	1.007	-0.284
	2/77	-2.2	1.5	0.991	-0.127
	4/77	-0.6	1.5	0.979	0.140
	1/78	-1.4	0.6	0.979	0.136
	2/78	-1.2	1.2	0.977	0.146
Composite	4/76	1.4	7.6	1.018	-0.049
	1/77	1.1	5.7	1.009	0.029
	2/77	1.1	11.0	1.033	-0.216
	4/77	-0.7	9.2	0.991	0.052
	1/78	-2.8	5.2	0.985	-0.179
	2/78	-2.1	9.0	0.999	-0.286

Weighing Survey

The second weighing survey was begun in November 1977, and was completed in March 1978. This survey was very similar to the one conducted during the first year of the Western Q.A. Program. Twelve sets of 3 weights, each containing a 1, 2, and 5g weight, slightly modified by filing away some of the mass, were cleaned and weighed several times on two different balances over a period of several days. Each balance was calibrated just prior to use with an NBS certified set of Class S weights. One set was sent to each participating agency, which then weighed the objects and reported results to Rockwell. The objects were reweighed after return to insure that no significant change had occurred during transit.

In the second survey, all agencies met the target range (± 1 mg) for all of their weighings. The sample range (± 0.5 mg) was met by 84% of all weighings made by all agencies. Thus the gross discrepancies observed in the weighings of three agencies, reported in the first annual report, have been eliminated, and there appears to be no weighing problems left. Table 8, taken from the second weighing survey report, summarizes the results from all the agencies.

A third weighing survey, which will include all groups in the East and West, is scheduled for early in 1979.

High Volume Flow Rate Measurement

The second high volume flow rate measurement survey was conducted in the second half of 1977. For this purpose, Rockwell submitted audit devices

TABLE 8. SUMMARY OF WEIGHING SURVEY

Agency Code[a]	Deviations From Rockwell (Nominal Value in mgx10)			Target Range[b]	Sample Range[b]
	1 gram	2 gram	5 gram		
A	-8	-1	-6	+++	+
B	3	2	-1	+++	+++
C	-1	1	-2	+++	+++
D	-5	-7	-6	+++	+
E_1	1	9	-4	+++	++
E_2	-2	-2	-4	+++	+++
F	3	2	-2	+++	+++
G	-4	-3	-2	+++	+++
H	-6	-2	-4	+++	++
I_1	-1	0	2	+++	+++
I_2	-1	0	1	+++	+++
J	-3	0	-2	+++	+++
K	-1	-1	-2	+++	+++
L	-2	0	-2	+++	+++
M	-4	-3	-6	+++	++
N	1	1	-4	+++	+++
O	6	5	1	+++	++

(a) Two entries from same agency indicates weighings using two different balances.

(b) Target range: $\pm$1 mg; Sample range: $\pm$0.5 mg. A (+) sign means weighing within designated range.

to participating agencies together with a test protocol. Agencies used the devices and at the same time performed a normal calibration. Agencies reported their measured flow rates as well as additional pressure and temperature data from which Rockwell calculated "true" flow rates. The results of the comparison between values measured by each agency and calculated by Rockwell are summarized in Table 9. This table gives each agency's calibration error, which is defined as the percent difference between the indicated flow and the "true" flow, measured at 1.132 m^3/min (40 ft^3/min).

A comparison between the first and second surveys indicated that substantial improvements in the calibration results had occurred, although some of the sites still showed large discrepancies. One problem that has not been resolved concerns the two agencies which use flow controllers. The audit device has been found to be inadequate when used in conjunction with the flow controllers, and no solution has yet been found for this problem.

In the two survey reports, it was suggested that a major problem in flow calibration was the lack of a definite, uniform set of instructions, issued by EPA, covering procedures, calculations, and data reporting. This deficiency has apparently been resolved by the publication of a comprehensive document entitled, "Investigation of Flow Rate Calibration Procedures Associated with the High Volume Method for Determination of Suspended Particulates" (2). This EPA document discusses in great detail the problems involved in calibrating high volume samplers, and recommends procedures for correcting and reporting all flow data to the same standard conditions of temperature and pressure. The document has been distributed to all the Western Q.A. participants, and hopefully additional improvement in the

TABLE 9. SUMMARY OF RESULTS IN THE SECOND HIGH VOLUME SURVEY

Agency	Site	Calibration Error, % Min.	1.132CMM	Max.	Remarks
A	1	2.4	12.3	17.5	Calibration poor, scatter excessive
B	1	2.7	4.8	13.2	Calibration fair
C	1	-0.8	-3.9	-7.4	Calibration good
D	1	3.2	5.5	10.2	Calibration fair
E	1	-1.9	0.1	+1.9	Calibration good
F	1	-2.5	-4.6	-7.1	Calibration good
G	1	-4.7	-7.3	-10.4	Calibration satisfactory
H	1				Flow controller employed, no meaningful result. See text.
I	1	6.3	8.4	8.8	Calibration fair
J	1				Flow controller employed, no meaningful result. See text.
K	1	-0.5	0.7	4.8	Calibration good
	2	0.0	3.6	9.6	Calibration good, one relatively bad point
	3	-1.4	0.2	3.9	Calibration good
L	1	-4.4	-5.7	-9.1	Calibration satisfactory
	2	-1.8	-5.9	-8.6	Calibration satisfactory
	3	-2.7	-2.7	18.1	Calibration poor, slope low, scatter high
	4	-5.3	-8.1	-14.0	Calibration fair, scatter high
	5	-6.5	-8.5	-12.5	Calibration fair, scatter high
M	1	-7.3	-8.2	-9.8	Calibration fair
	2	-2.4	-5.2	-10.7	Calibration good, except for one bad point
	3	2.8	-0.9	8.7	Calibration satisfactory, scatter high
	4	-7.1	-8.0	-9.4	Calibration fair
	5	-7.5	-9.1	-13.7	Calibration satisfactory
	6	-6.1	-8.6	-14.8	Calibration satisfactory
	7	-3.1	-5.1	-8.9	Calibration good

(continued)

TABLE 9 (Continued)

Agency	Site	Calibration Error, % Min.	1.132CMM	Max.	Remarks
N	1	-0.3	-1.0	-1.8	Calibration good
	2	6.7	7.6	12.8	Calibration poor, only four points
	3	-6.4	-7.2	-7.6	Calibration fair
	4	-1.4	-4.4	-5.1	Calibration good
	5	1.1	-1.5	-3.4	Calibration good
	6	-7.7	-9.4	-11.7	Calibration fair
	7	3.8	5.5	7.1	Calibration fair
	8	-4.9	-8.7	-14.8	Calibration poor, high scatter
	9	4.5	7.4	16.0	Calibration poor
	10	27.0	17.8	47.6	Calibration poor, high scatter
O	1	-3.2	-3.8	-5.4	Calibration good
P	1	-3.8	-5.1	-9.6	Calibration satisfactory
	2	-5.6	-6.8	-12.8	Calibration fair
	3	-1.0	-2.6	-4.2	Calibration good
	4	-1.8	0.9	6.9	Calibration good, except for high scatter

results will show up in the third survey which is scheduled for the beginning of 1979.

A pertinent observation concerning the new publication may be offered here. The document appears to be somewhat difficult for the non-expert, particularly because of the necessarily complex notation. However, it is understood that the EPA is planning to conduct special workshops to discuss and review the contents of the document with operators and other individuals responsible for implementing the recommendations in the book.

Interlaboratory Comparison Study of Metal Samples

One of the requirements of the Western Quality Assurance Program is to duplicate approximately 10% of the analyses performed by a designated laboratory (denoted here as Laboratory A) for metals collected with particulates in high-volume samplers. The method of analysis is atomic absorption, and the elements that were originally listed included Be, Ca, B, Zn, Cd, Cr, Co, Cu, Fe, Pb, Mn, Mo and Ni.

During the first year of the program several filter batches analyzed both by Laboratory A and by Rockwell disclosed very large discrepancies between the analyses by two laboratories, and no assignable cause could be determined. In order to investigate the origin(s) of the differences, a test program was designed and initiated during September 1977. The test consisted of a sample exchange program in which synthetic metal samples prepared by Rockwell were analyzed by both Laboratory A and the Rockwell Chemistry Laboratory. Three types of samples were chosen in order to investigate three general sources of discrepancies: contamination, extraction,

and analytical procedures. The samples included were as follows:

1. Uncut blank filters.
2. Spiked filters containing known concentrations of Cd, Co, Cr, Cu, Fe, Mn, Mo, Ni, and Pb. The concentrations were chosen to be significantly above detection limits.
3. Solutions of the above metals dissolved in 10% HNO_3.

In addition to the above analyses, all of the liquid extracts analyzed by Laboratory A were returned to Rockwell for reanalysis.

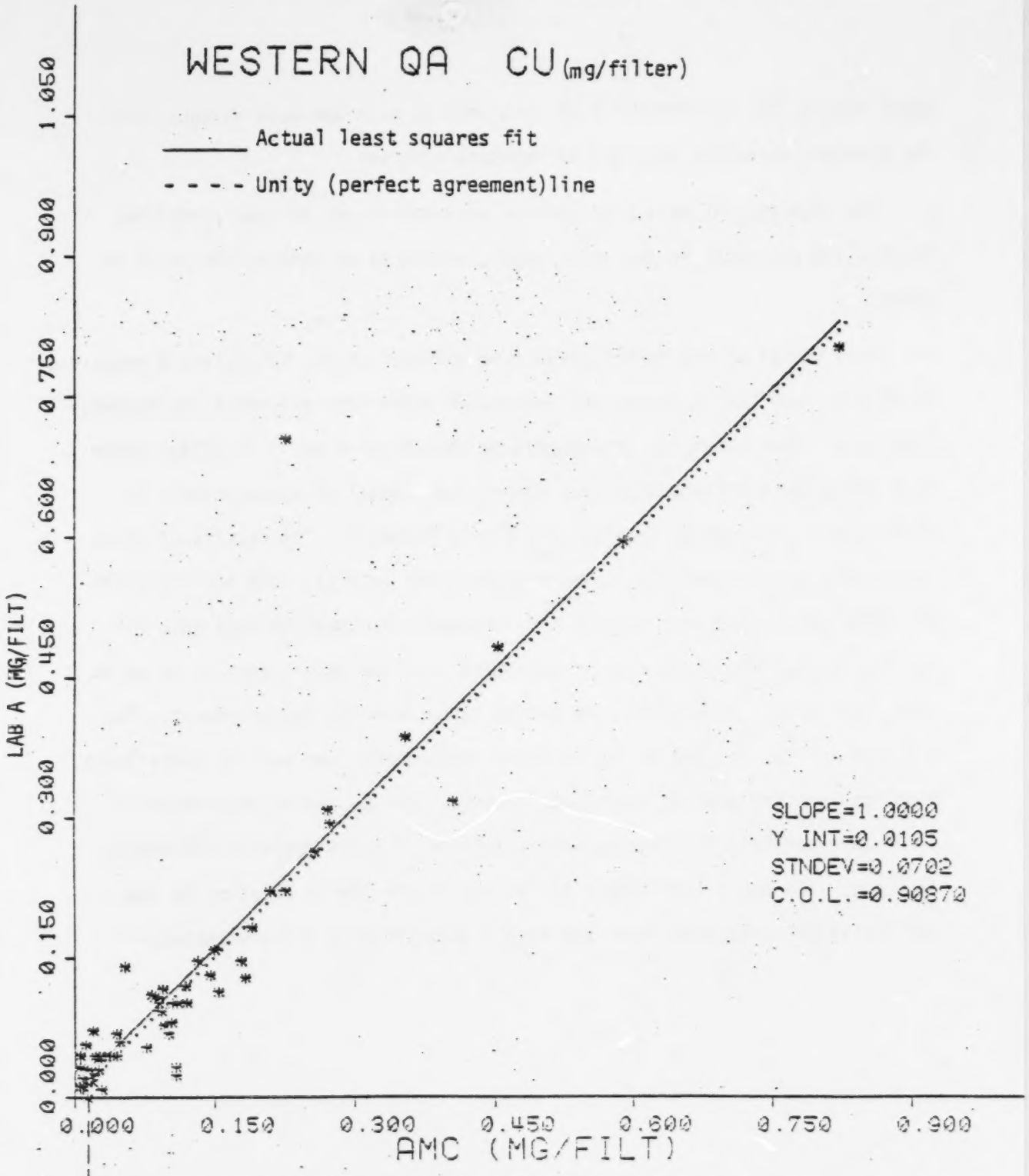
The results of this joint testing program were reported to EPA in a long, detailed report dated January 30, 1978. The principal conclusion resulting from the study was that the discrepancies between the two laboratories could be ascribed primarily to differences in extraction procedures. Laboratory A extracted metal samples by refluxing the filter samples with a mixture of nitric acid and hydrochloric acid, and then diluting the concentrate with 50% HCl. Rockwell, on the other hand, extracted filter samples with 10% HNO_3 and diluted the concentrate also with 10% HNO_3. For most metals, the study disclosed that the 10% HNO_3 procedure gave good accuracy and precision in the hands of both Rockwell and Laboratory A, and that the results were definitely superior to those obtained by means of the 50% HCl procedure. The low percentage recoveries by Laboratory A in past studies could be reasonably explained by the relatively poor recovery efficiency obtained from the 50% HCl extraction procedure.

For the analysis of Mo and Cr, the 10% HNO_3 extraction procedure had to be modified, however, in order to obtain accurate AA analyses. For both of

these metals, the addition of 0.5% lanthanum to both the acid extracts and the standard solutions resulted in improved accuracy.

The high and variable blank values obtained in the earlier comparison studies did not occur in the joint study, and hence no conclusions could be drawn.

As a result of the interlaboratory comparison study, Laboratory A requested permission from EPA to change the extraction procedures and adopt the Rockwell procedure. The change was implemented by Laboratory A early in 1978. Since that time, three different batches comprising a total of approximately 74 filters were analyzed by Laboratory A and by Rockwell. The results of these comparisons were summarized in two reports dated June 13, 1978 and September 25, 1978, which have been sent to EPA. Comparisons could be made only for Cu, Fe, Mn, and Pb, since the concentration of other metals were at or below detection limit. Figures 2-5 are graphs taken from the latter report. The analyses for Cu, Fe, and Mn showed major improvement over earlier comparisons. High data scatter remains a problem, however. For Cu and Fe deposition levels are sufficiently high so that precision of ± 10% could be reasonably expected. The deposition levels for Mn and Pb are low in relation to the 10% analytical precision level and high data scatter is to be expected.



(I) Exposure level at and above which Rockwell can analyze with better than ±10% precision.

Figure 2. Split sample analyses for Cu in hi-vol filters - Comparison between Laboratory A and Rockwell

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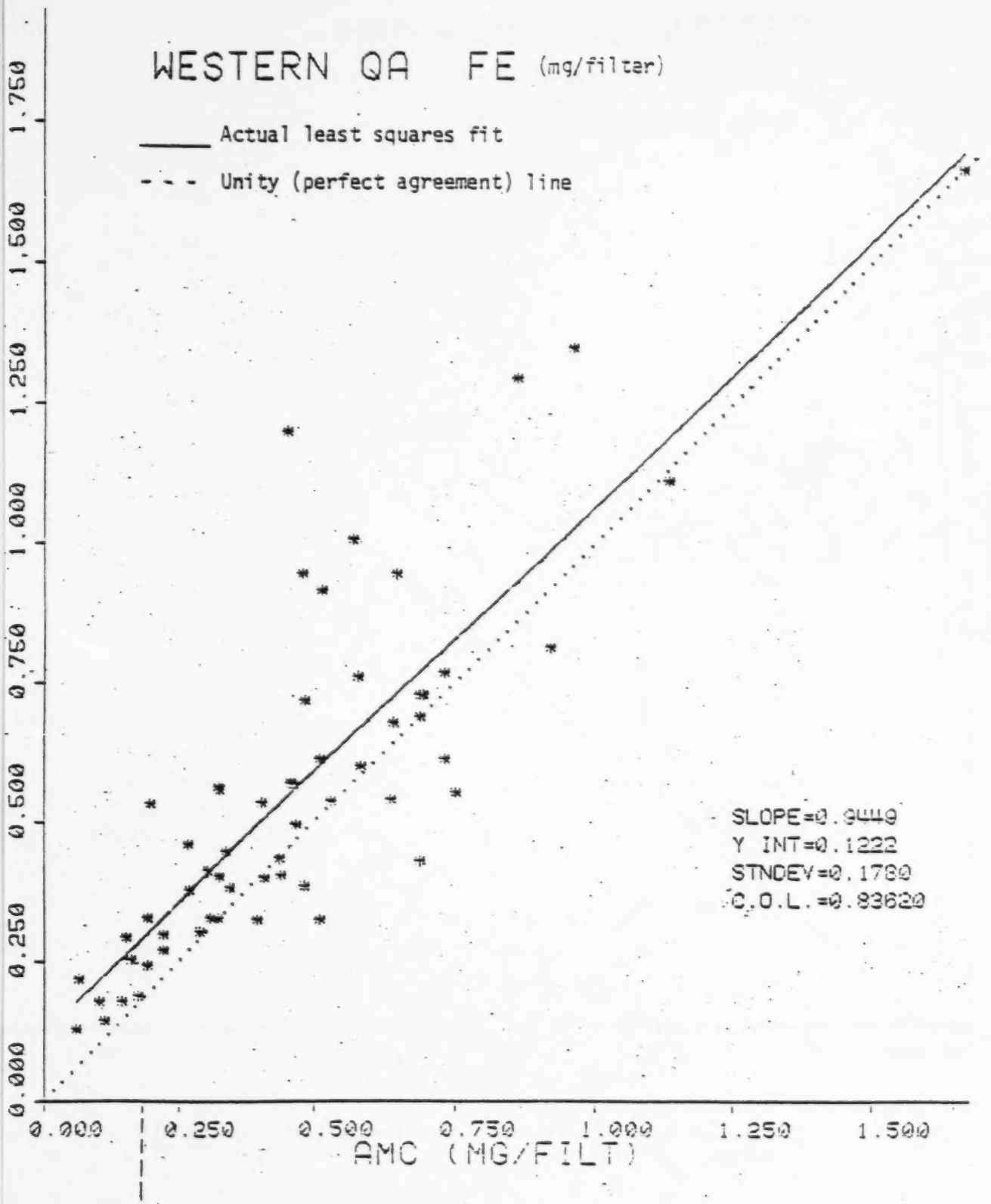

(1) Exposure level at and above which Rockwell can analyze with better than ±10% precision.

Figure 3. Split sample analysis for Fe in hi-vol filters - Comparison between Laboratory A and Rockwell

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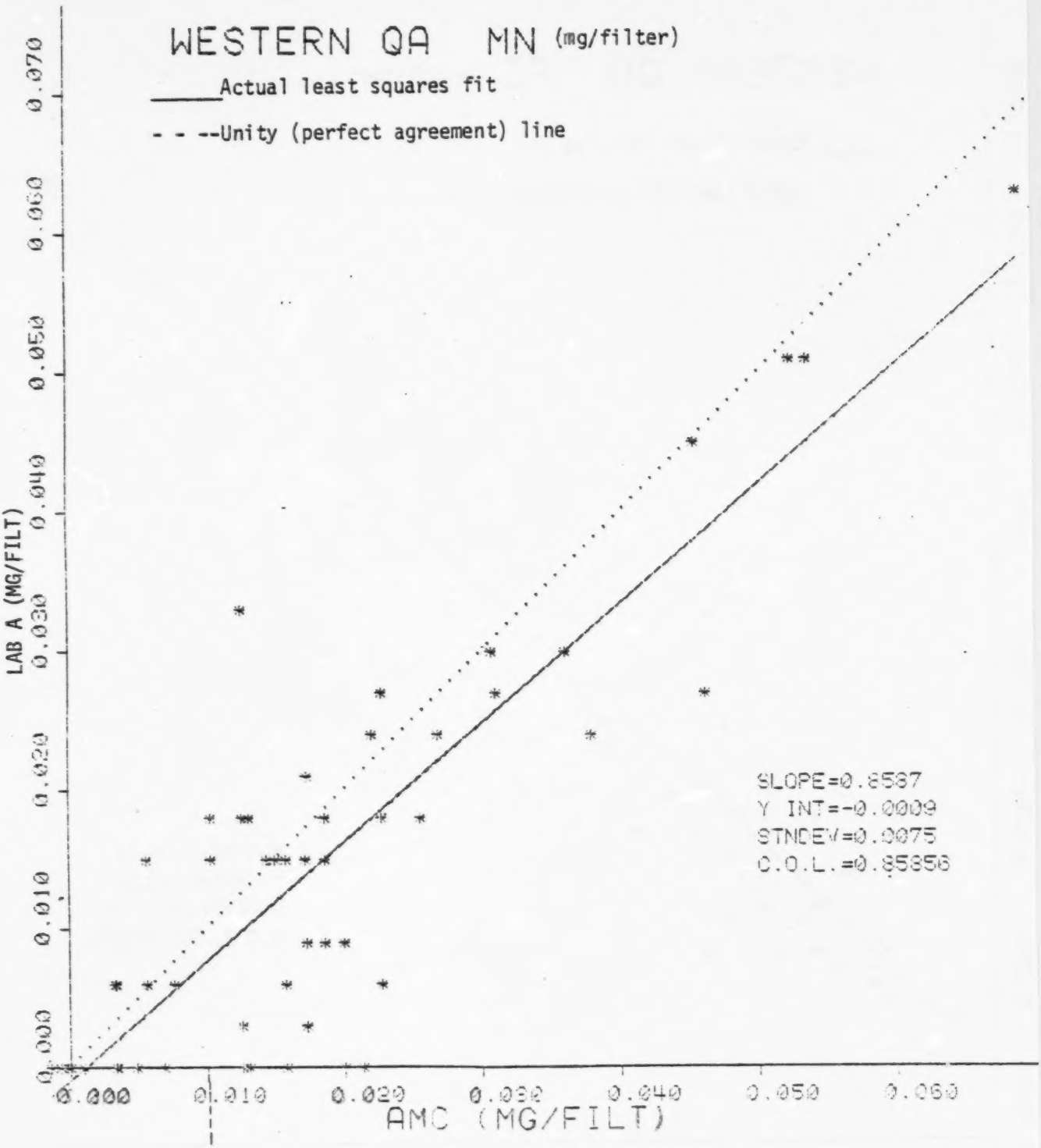

(1) Exposure level at and above which Rockwell can analyze with better than ± 10% precision.

Figure 4. Split sample analysis for Mn in hi-vol filters. Comparison between Laboratory A and Rockwell.

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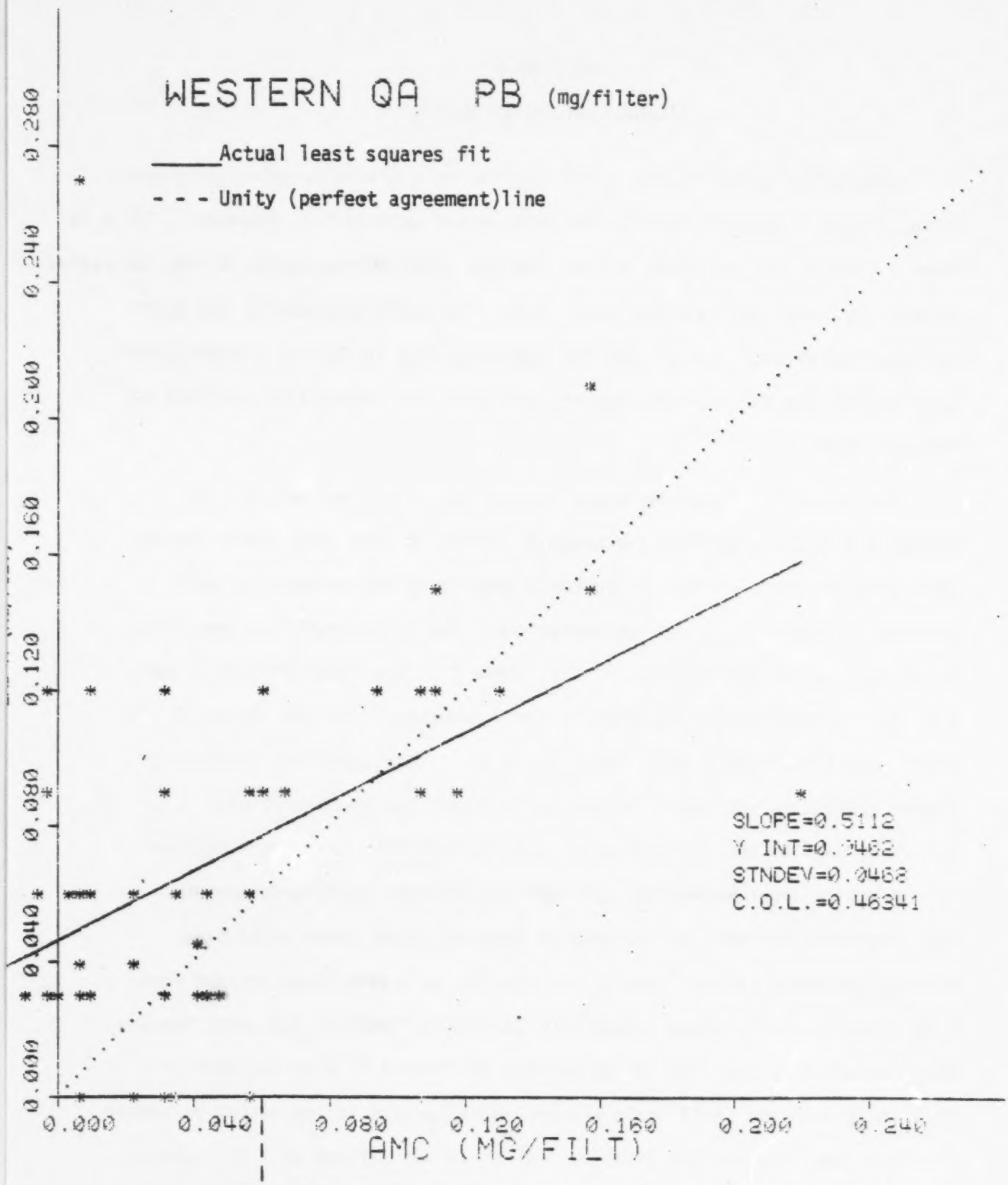

(1) Exposure level at and above which Rockwell can analyze with better than ±10% precision.

Figure 5. Split sample analysis for Pb in hi-vol filters. Comparison between Laboratory A and Rockwell.

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SECTION 4

CALIBRATION SYSTEM AUDITS

Calibration System Audits of air monitoring stations operated by participating groups is a major task in the Western and Eastern Q.A. Programs. Table 10 shows a list of all the sites audited and the dates of the audits during the period between September 1977 and September 1978. The audit procedures, the audit devices, operational checks, and the standards used in the field audits were described in the first annual report, and hence the information will not be repeated here.

The format for reporting audit results has undergone considerable change during the course of the program. Prior to June 1978, audit results were given in terms of linear constants describing the response of each analyzer to known pollutant concentrations. The relationship was described by an equation of the form $C = A + BX$, where C is the concentration in ppm, X is the analyzer output measured in some convenient form (mv, volts, % chart, chart divisions, etc.) and A and B are linear constants obtained by linear regression analysis. In the audit report two pairs of linear constants were tabulated, namely the pair derived from the latest station multipoint calibration and the pair derived from the audit measurements. The comparison between the two sets of data was shown graphically by drawing the two straight lines (i.e., station and audit lines) on the same C vs X graph. Differences between the station calibration and audit results were evaluated by calculating the percent difference in C at the nominal full scale output. (Full scale was defined according to the output parameter in use at the station; for example, full scale was defined as 1 volt output

TABLE 10. SUMMARY OF AUDITS PERFORMED BY ROCKWELL DURING THE PERIOD SEPTEMBER 1977 TO SEPTEMBER 1978

Agency	Site	Date
C-a	1	Nov.1977; March 1978; June 1978; Sept.1978
C-b	023	Nov.1977; March 1978; June 1978; Sept.1978
State of Colorado	CARIH	Oct.1977; Feb.1978; May 1978; Aug. 1978
	CAMP	Oct.1977; Feb.1978; May 1978; Aug. 1978
	Greeley	Oct.1977; Feb.1978; May 1978; Aug. 1978
	Arvada	Oct.1977; Feb.1978; May 1978
	Colorado Springs	Oct.1977; Feb.1978; May 1978; Aug. 1978
	Welby	Oct.1977; Feb.1978; May 1978; Aug. 1978
	Grand Junction	Nov.1977; March 1978
EPA/Custer, MT	Custer	May 1978
EPA/EMSL-RTP	RTP	Nov.1977; March 1978; May 1978; Aug.1978
EPA/EMSL (Northrop Services Inc.)	Las Vegas	Dec.1977; April 1978; July 1978
EPA/Corvallis (Pacific Northwest Laboratory)	Hae Koolie	Jan.1978; April 1978; July 1978
	B&W	July 1978
LLL/Imperial Valley	2	Dec.1977
	3	Dec.1977
	6	Dec.1977; April 1978
	Mobile	April 1978
State of Montana	Billings	Jan.1978; April 1978; Aug.1978
	Laurel	April 1978
	Hwy Junction	Jan.1978; April 1978; Aug.1978

(continued)

TABLE 10. (Continued)

Agency	Site	Date
State of Montana (continued)	Millcreek	Jan.1978
	Broudy	Jan.1978; April 1978
	Microwave	Jan.1978; April 1978
	Hebgen Park	April 1978; Aug.1978
	Lions Park	April 1978; Aug.1978
	Alpine West	Jan.1978; April 1978
	Lincoln School	April 1978; Aug.1978
	Malfunction Junction	April 1978
State of New Mexico	Reservation	Dec.1977; March 1978; June 1978; Sept.1978
	Substation	Dec.1977; March 1978; June 1978; Sept.1978
	Water Tank	Dec.1977; March 1978; June 1978; Sept.1978
	State Calib.	Sept.1978
NOAA	Boulder	May 1978
State of North Dakota	Stanton	Jan.1978; April 1978; July 1978
	Bismarck	Jan.1978; April 1978; July 1978
U_a/U_b	A6	Dec.1977; March 1978; June 1978; Sept.1978
State of Utah	Price	Jan.1978; April 1978; July 1978
	Huntington	Jan.1978; April 1978; July 1978
	Bountiful	Jan.1978; April 1978; July 1978
	Provo	Jan.1978; April 1978; July 1978
	Magna	Jan.1978; April 1978; July 1978
	Ogden	Jan.1978; April 1978; July 1978
	Cedar City I	Nov.1977; June 1978
	Cedar City II	April 1978; June 1978; Sept. 1978

(continued)

TABLE 10. (Continued)

Agency	Site	Date
State of Utah (continued)	Cedar City III	Nov.1977; April 1978
	State Calib.	Jan.1978; April 1978; July 1978
Woodward-Clyde	Roosevelt Hot Springs	June 1978; Sept.1978
State of Wyoming	Patrick Draw	Dec.1977; March 1978; June 1978; Sept.1978

or as 100 chart divisions, etc.). Since in this reporting format the magnitude of B depends on the units of the output parameter, comparison of calibration constants among different instruments and agencies was difficult.

At the request of EPA a second method was adopted for reporting audit results, starting with audits performed after June 1978. The second method compares the concentrations delivered to the analyzer from the audit device to those predicted from the observed instrumental response and the latest station multipoint calibration. Mathematically, the results are expressed in the form $C_2 = \alpha + \beta C_1$, where C_2 is the station concentration, C_1 is the audit concentration, β is the slope, and α is the intercept. Perfect agreement between station and audit is represented by the "unity line" $\beta = 1$, $\alpha = 0$. Audit reports show graphs of C_2 vs C_1, and the values of α and β are given in tabular form. The advantage of the second method is that β is non-dimensional, and its value is independent of the analyzer range and the units used to express analyzer output.

The percent difference between station concentrations are readily expressed in terms of the magnitude of α and β by means of the equation

$$\% \text{ diff} = 100 \times \frac{C_2 - C_1}{C_1} = 100 \left[(\beta - 1) + \frac{\alpha}{C_1} \right] \qquad (2)$$

In order to evaluate the station performance the percent difference is calculated at the nominal full scale of the analyzer (for example: $C_1 = 0.5$ ppm for most NO analyzers). Since normally the magnitude of α/C_1 is small compared to $\beta - 1$, the percent difference is approximately a constant represented by the quantity $100 (\beta - 1)$. It is this latter relationship which makes β a useful comparison parameter.

Although the two methods for evaluating performance appear to give different measures of comparison, it has been shown in previous audit reports that the two methods are in fact equivalent.

In August 1978, EPA published a set of proposed quality assurance regulations under EPA 40 CFR Part 58, "Air Quality Surveillance and Data Reporting;" Appendix A - Quality Assurance Requirements for State and Local Air Monitoring Stations (SLAMS) (1). The proposed regulations describe still a third method for evaluating calibration performance. The regulation requires calculating the percent difference d_i for each audit point, and then computing the average and standard deviation for all the d_i's. The average percent difference $\bar{d}_j$ and the standard deviation S_j are used to estimate the upper and lower probability limits (measured as a percent) which comprises 95% of all the audit data and can be used to predict where future measurements are expected to lie. The calculations outlined in Appendix A have been applied to audits carried out since August 1978. (It should be noted that Appendix A is expected to be revised in the near future, and we understand that the use of $\bar{d}_j$ and S_j for calibration system audits will be dropped.) The audit reports since August 1978 include tables of $\bar{d}_j$ and S_j, but no attempts have been made to interpret these data.

During the first two years of the program, over 150 Calibration System Audits have been performed; hence, a very large amount of data has been collected. Seven quarterly audits have been performed in the EPA/EMSL laboratory at Research Triangle Park (RTP), N.C. for the purpose of comparing standards and procedures between Rockwell and EPA. The results of these special audits have been very satisfactory as the differences between the two laboratories have been generally 5% or less.

Table II shows a summary of overall performance for each type of pollutant being audited. The table shows the total number of audits, the average percent error (calculated as described earlier in Section 4 buth without regard to

TABLE II. SUMMARY OF CALIBRATION SYSTEM AUDIT RESULTS

Pollutant	No. of Audits	Avg. Percent Error	Percent of Audits With Error of 10% or Less
CO	73	6.0	86
CH_4/THC	63	8.5	75
NO_x	78	11.4	63
NO	90	12.3	64
NO_2	69	11.5	67
SO_2	151	12.0	64
O_3	108	12.9	53

sign) and the percent of the audits for which the calibration error is 10% or less. The 10% criterion has been adopted as an arbitrary but reasonable measure of "satisfactory" performance. The last column in Table 11 gives a good qualitative indication of the relative calibration accuracy for the various pollutants and the results clearly reflect the relative complexity of the calibration methods.

Figure 6 presents some evidence on the impact of the QA program on the quality of the data generated by the participating groups. Figure 6 shows a remarkable improvement in the calibration accuracy for NO, NO_x, and NO_2. For the first two quarters, the observed average errors were in excess of 20%. In the last quarter, the average errors decreased to approximately 5%. For SO_2 a somewhat different pattern is observed. During the first two quarters the average error was greater than 20%. Several groups displayed large calibration differences caused by errors in procedure and/or calculation. After these large errors were eliminated, the average percent errors have remained essentially constant at about 10%. This may well be the ultimate limit that can be expected for the current calibration methods.

The average percent error for CO has been approximately 5% since the second quarter. This is excellent agreement and it clearly indicates that calibration of CO presents no difficulties. This conclusion is also consistent with the results of the interlaboratory CO performance survey described in Section 3.0. It is thus evident that CO data generated by the western groups is of relatively high quality. The situation for CH_4/THC is comparable although not quite as good as CO, even though a gradual improvement in audit results might appear to have occurred. The maximum observed in the second quarter

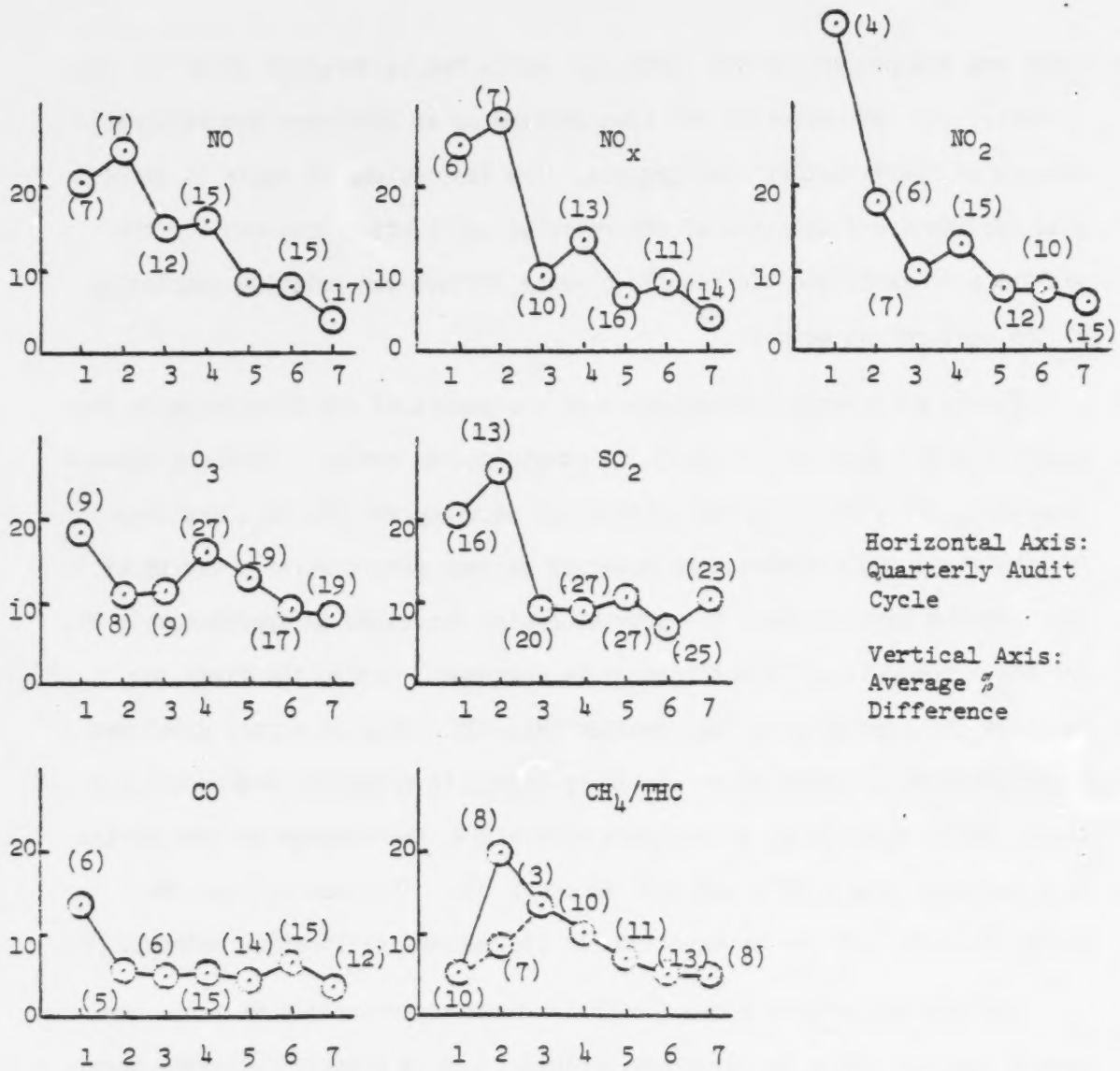

Figure 6. Average calibration error vs audit cycle. Numbers in parentheses indicate total number of audits per quarter. The seven quarters above extend from the first quarter of 1977 through the third quarter in 1978.

is attributed to a single audit result in which a very large calibration error (subsequently found to have been caused by improper calibration procedure) unduly affected the average. If this non-typical station were removed from the average, the observed pattern would be as shown by the dotted line. The relatively high error in the third quarter has to be viewed with caution since the average is made up of only three audits.

The most significant calibration problem encountered in the program is that of ozone. A small downward trend in the calibration error may have occurred, but compared to the other pollutants the improvement is small and the average percent error has remained at or above 10%. Of course, the major problem in the calibration of ozone is that there is no stable, reliable standard readily adaptable for field use. EPA has recognized this problem and has recently issued a number of documents in draft form (3,4) to help monitoring groups in improving the ozone calibration procedures. In addition, a recent proposed EPA change (5) designates UV photometry as the new reference method for ozone calibration. We expect the ozone data to begin showing improved accuracy as more groups adopt this simpler calibration procedure.

SECTION 5

TECHNICAL ASSISTANCE

In accordance with the provisions of the contract, Rockwell has provided technical assistance to the various participating agencies as requested by the Project Officer. Some of the assistance has been relatively minor, and consisted of telephone discussion of various agency problems, citing of literature references or help with procedural difficulties.

Much technical assistance is given informally during site evaluations and particularly during field audit visits where the exchange of ideas and information between Rockwell and Agency personnel is oftentimes extensive and intensive.

The following is a list of interactions with agencies during which Rockwell provided technical assistance.

Agency A - Traceability and certification were established for three CO cylinders sent to Rockwell by this agency. Notes and advice on the vapor pressure correction used with a Hastings Bubblemeter Kit with flows over 1 liter/min were also supplied. The correction factor given is the one used by Rockwell during field service (November 1977).

Agency B - Several meetings and discussions were conducted with this agency in an effort to resolve problems encountered during an interlaboratory sample survey for $SO_4^=$. No resolution to the problem was readily apparent although the fact that the agency used the $BaCl_2$ method for $SO_4^=$ as opposed to the more accurate MTB (methylthymol blue) method may be involved. A full explanation of the interlaboratory comparison methods at each of the sample

concentrations levels was also presented to this agency at a meeting held at the agency's headquarters (December 1977).

Agency C - This agency requested certification of the SO_2 permeation rate and the ppm concentration for an NO cylinder. Traceability was to be established to NBS materials but the agency standards have yet to arrive (January 1978).

Agency D - This agency sought specific assistance with an $SO_4^=$-NO_3^- problem. While the agency had no problem analyzing liquid samples, there was some question as to the effectiveness of the ultrasonic extraction procedure currently in use. It was suggested that filters spiked with known amounts of material be extracted as per normal to resolve this problem (February 1978).

Agency E - Rockwell personnel visited this agency in April 1978 subsequent to a request for technical assistance with the NO_3^-, $SO_4^=$ and As analyses. The instrumentation used for this analysis was functioning correctly. Sample preparation was cited as a possible cause for the earlier problem seen with the NO_3^- analysis.

In April 1978, three cylinder standards (CO, NO and CH_4) were certified by Rockwell and traceability to NBS supplied.

An extensive split-sample program was conducted with this agency for the analysis of heavy metals on high volume filters. Approximately 74 agency filters were analyzed by Rockwell between December 1977 and June 1978 for Al, As, Cd, Cu, Fe, Pb, and Zn. In the earlier analyses the indicated agreement between the two laboratories was good for Cd, Cu, and Pb, fair for As, and poor for Al, Fe and Zn. The comparisons completed in June 1978

showed remarkable improvement in the analyses of Fe and Zn. The As comparison also showed improvement, possibly as a result of the implementation by the agency of the more precise automated-flameless technique used by Rockwell. Some differences in the Al analysis still remain.

Agency F - An extensive split-sample program was conducted with this agency for the analysis of heavy metals on hi-volume filters. Presumably identical hi-vol filter strips were analyzed for SB, As, Be, Cd, Cu, Pb, Hg, Se, and Zn by the Rockwell Chemistry group and by the agency laboratory. Problems were seen with the Cd, Hg, and Zn results while those for Pb and Cu were relatively good (November 1977 - May 1978). Later in the year (May 1978) this agency had difficulties during an interlaboratory survey and requested replicate samples. The problems experienced with $SO_4^=$ were traced to a defective absorption solution.

Agency G - Two cylinders of NO and CO were shipped to Rockwell to establish NBS traceability and absolute concentration values. The purpose of this task was to resolve a problem concerning the accuracy of the vendor's analysis. Results were sent to the agency personnel as well as to the Project Officer (September 1978).

Agency H - Eight cylinders were sent to Rockwell for certification and to establish NBS traceability. All were methane cylinders containing approximately 9-16 ppm CH_4. The results of the analyses were sent to the agency (September 1978).

SECTION 6

PLANS FOR NEXT YEAR

The general plans for next year are to continue the current program of Quality Control Reference Sample Audits, Calibration System Audits and technical assistance. A major addition, of course, is the implementation of the Eastern Q.A. Program. During the next year the five or six groups which have not yet been evaluated will have to be visited, and the evaluation reports for all the Eastern Q.A. groups will be completed. Quality Control Reference Sample Audits and Calibration System Audits will be expanded to include the participation of the eastern groups. While the need for technical assistance in the west may be expected to decline, the requirements in the east are difficult to estimate.

With the implementation of the Appendix A guidelines, some additional effort may be required towards modification of existing audit procedures, particularly with respect to ozone. The calculation procedures in Appendix A are likely to be changed by EPA during the next year, and therefore the reporting format may have to be modified once more. Improved methods for evaluating individual and overall performance as a function of time should be developed, and some additional statistical analysis may be desirable. The use of the computer for storing and analyzing Calibration System Audit data should be made a high priority task.

As indicated in the first annual report, since the ultimate purpose of the program is to help the participating groups in improving the quality of their data base, some modifications in the program may become desirable as the collective experience of all groups increases. Thus constant reevaluation of the program is in order, and changes will be made whenever they

seem appropriate, given the consent and cooperation of the Project Officer and all interested parties.

REFERENCES

1. Environmental Protection Agency; Proposed Regulatory Revision to 40 CRF, Part 58 (Appendix A), "Air Quality Surveillance and Data Reporting," Federal Register, Vol. 43, August 7, 1978, p 34906.

2. Environmental Protection Agency; "Investigation of Flow Rate Calibration Procedures Associated with the High Volume Method for Determination of Suspended Particulates," EPA-600/4-78-047, August 1978.

3. Environmental Protection Agency; "Technical Assistance Document for the Calibration of Ambient Ozone Monitors," available from EPA, Department E (MD-76), Research Triangle Park, N.C. 27711.

4. Environmental Protection Agency; "Transfer Standards for Calibration of Ambient Air Monitoring Analyzers for Ozone," EPA Publication available in draft form from EPA, Department E (MD-76), Research Triangle Park, N.C. 27711.

5. Environmental Protection Agency; Proposed Amendments to 40 CRF Part 50 (Appendix D), "Measurement of Ozone in the Atmosphere," Federal Register, Vol. 43, June 22, 1978, p 26971.